

~~Authorized Signature~~

**TITLE 47
LEGISLATIVE RULE
DEPARTMENT OF ENVIRONMENTAL PROTECTION
WATER RESOURCES**

**SERIES 32
ENVIRONMENTAL LABORATORIES
CERTIFICATION AND STANDARDS OF PERFORMANCE**

FILED

2009 MAY 12 PM 3:59

OFFICE WEST VIRGINIA
SECRETARY OF STATE

§47-32-1. General.

1.1. Scope. -- This rule governs the certification of laboratories conducting environmental analysis of waste and wastewater performed as required by rules or orders issued pursuant to the covered statutory programs. The rule establishes the provisions for obtaining and maintaining laboratory certifications and the criteria and procedures laboratories will be required to follow in analyzing samples.

1.2. Authority. -- W. Va. Code §22-1-15.

1.3. Filing Date. -- May 12, 2009

1.4. Effective Date. -- July 1, 2009

1.5. Incorporation by Reference. -- The Department hereby adopts and incorporates into this rule the approved "Guidelines Establishing Test Procedures for the Analysis of Pollutants" 40 CFR 136, EPA SW 846 Methods, and such other methods as may be approved by U.S. Environmental Protection Agency (EPA) or the Secretary.

1.6. Construction. -- This rule shall be liberally construed to permit the Department to discharge its statutory functions and to effectuate the purposes of the laboratory certification program.

1.7. Purpose of this Rule. -- This rule is promulgated to ensure that the results of environmental analyses are accurate, reproducible and verifiable. This purpose will be achieved by:

1.7.1. Establishing the administrative procedures to be followed by certified laboratories and laboratories seeking certification;

1.7.2. Establishing the categories in which, and the parameters for which laboratories may be certified;

1.7.3. Establishing the minimum requirements, criteria and procedures for laboratory equipment and supplies, practices, methodology, quality control, personnel, facilities, data reporting, and laboratory and record maintenance, which a certified laboratory shall continually meet; and

1.7.4. Establishing the enforcement procedures the Department will follow to ensure that all certified laboratories or laboratories seeking certification are in compliance with this rule.

1.8. Certification Program Requirements.

1.8.1. A laboratory analyzing samples for compliance with adopted rules, permits, or orders issued

pursuant to a covered statutory program will follow the procedures set forth in this rule in order to obtain and maintain certification. The provisions of this rule are only applicable to tests required by State and Federal regulatory programs.

1.8.2. Certified laboratories and laboratories seeking certification will analyze all samples requiring testing under this rule in accordance with the procedures and methods required by this rule.

1.9. Program Information and Communications. — Questions concerning the requirements of this rule should be directed to the Department of Environmental Protection, Division of Water and Waste Management, Quality Assurance Program, 601 57th Street SE, Charleston, WV 25304-2345.

§47-32-2. Definitions.

The following words and terms, when used in this rule have the following meanings unless the context clearly indicates otherwise.

2.1. "Accredited" means an approval conferred upon institutions or programs where appropriate by a nationally recognized accrediting agency or association as determined by the Department.

2.2. "Accuracy" means the closeness of agreement between an observed value and the accepted reference value. Accuracy is best determined through the analyses of a sample spiked with a known concentration of target analytes and this value compared to an unspiked aliquot.

2.3. "Analyte" means an element, ion, isotope, compound, or component of interest to the analyst.

2.4. "Analytical Reagent Grade" (AR), "ACS reagent grade", and "Reagent Grade" are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

2.5. "Analyst" means the individual who performs the analytical methods and associated techniques and who is responsible for applying the required laboratory practices and quality controls to meet the required level of quality.

2.6. "APHA Standard Methods" or "Standard Methods for the Examination of Water and Wastewater" means the methods published by the American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005.

2.7. "Approved analytical methods" are those analytical or test methods cited in the Code of Federal Regulations as being approved by EPA or such other methods as shall be approved by the Secretary.

2.8. "Batch" means the environmental samples that are prepared or analyzed together using the same procedures, personnel, lots of reagents, and standards.

2.9. "Batch, Analytical" means a batch composed of prepared environmental samples that are analyzed together as a group. An analytical batch may contain samples originating from various environmental matrices and can exceed 20 samples.

2.10. "Batch, Preparation" means a batch composed of 1 to 20 environmental samples of the same matrix

with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours.

2.11. "Cancellation" means the voluntary removal of a previously certified laboratory from the laboratory certification program.

2.12. "Category" means a group of parameters for which certification is offered.

2.13. "Certification" means the approval granted by the Secretary authorizing a laboratory to provide environmental compliance data.

2.14. "Certification parameter" means a parameter that is identified in a proficiency test sample and that is used to evaluate the overall analytical performance of a laboratory on the specific method.

2.15. "Certification year" is that period of time following the date upon which the laboratory first receives certification for any parameter or category and lasting for 365 consecutive days.

2.16. "Certified thermometer" is a thermometer that has documentation from the manufacturer showing that it has been compared against a National Institute for Standards Testing (NIST) thermometer covering the temperature ranges employed by the laboratory.

2.17. "CFR" means the Code of Federal Regulations.

2.18. "Compliance analysis" means the analysis of a sample that is required to be analyzed by a Department rule, permit or order.

2.19. "Covered statutory programs" means one of the regulatory programs developed under statutory authority of one of the following acts of the Legislature:

2.19.1. Water Pollution Control Act, WV Code §22-11-1.

2.19.2. Hazardous Waste Management Act, WV Code §22-18-1.

2.19.3. Hazardous Waste Emergency Response Fund Act, WV Code §22-19-1.

2.19.4. Underground Storage Tank Act, WV Code §22-17-1.

2.19.5. Solid Waste Management Act, WV Code §22-15-1.

2.19.6. Groundwater Protection Act, WV Code §22-12-1.

2.20. "Deficiency" means a deviation from acceptable procedures or practices.

2.21. "Department" means the West Virginia Department of Environmental Protection.

2.22. "EPA" and "USEPA" means the United States Environmental Protection Agency.

2.23. "Laboratory" means a facility conducting tests or analyses of parameters for which certification is required, where the results of such tests or analyses are used for purposes of demonstrating compliance under the covered statutory programs. Provided; The term "laboratory" shall not include individuals conducting analyses of constituents that must be conducted in the field because of practical constraints; such as, but not

limited to pH, dissolved oxygen, total residual chlorine and sulfide.

2.24. "Laboratory pure water" means distilled or deionized water which is free of contaminants that interfere with analytical tests.

2.25. "Laboratory seeking certification" means an uncertified laboratory which has submitted an acceptable application and the appropriate fee.

2.26. "List of certified parameters" means the document displaying the categories and parameters for which a laboratory is certified.

2.27. "Matrix or matrices" means the media of an environmental sample, either non-potable water or solid and chemical materials.

2.28. "Method" means the scientific technique used to perform testing or analyses of an environmental sample.

2.29. "Mobile laboratory" means a portable enclosed structure within which testing or analyses of environmental samples occurs.

2.30. "NPDES" means National Pollutant Discharge Elimination System.

2.31. "Nonpotable water" means wastewater, ambient water, surface water, groundwater, effluents, water treatment chemicals, and toxicity characteristic leaching procedure or other extracts.

2.32. "Parameter" means an analytical method or test within a category and for which certification is offered.

2.33. "Proficiency test sample" means a sample containing a known amount of a specific or combination of parameters used in part to evaluate the performance of a laboratory.

2.34. "Person, Persons, or applicant" means any industrial user, public or private corporation, institution, association, firm or company organized or existing under the laws of this or any other state or country; state of West Virginia; governmental agency, including federal facilities; political subdivision; county commission; municipal corporation; industry; sanitary district; public service district; drainage district; soil conservation district; watershed improvement district; partnership; trust; estate; person or individual; group of persons or individuals acting individually or as a group; or any legal entity whatever.

2.35. "Personal and direct supervision" means that a supervisor is available either in person or on call at all times when laboratory procedures are being performed.

2.36. "Precision" means the agreement among a set of measurements performed on duplicate samples without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses.

2.37. "Quality Assurance Program" means a program developed to achieve the purposes of subsection 1.7 for the covered statutory programs of the Department.

2.38. "Quality Manual" means the document stating, or making reference to the policies, objectives, principles, responsibilities, accountability, implementation plans, methods, operation procedures, or other

documents of an environmental laboratory for ensuring the quality of its testing analyses.

2.39. "Raw Data" means that data acquired in the process of collecting and analyzing samples for compliance testing purposes. Raw data includes such sampling report forms, sample log books, laboratory bench sheets, calculations and formulas, and analytical data and notes as are used during sample analysis. Raw data may be in the form of graphs, line recorder charts, handwritten data, or computer printouts made at or near the time of the analysis or sample collection.

2.40. "Revocation of certification" means the action taken by the Department to halt the certification of a laboratory for cause.

2.41. "Sample Duplicate" means a sample prepared by dividing a homogeneous sample into separate parts so that each part is also homogeneous and representative of the original sample.

2.42. "Secretary" means the Secretary of the West Virginia Department of Environmental Protection or his or her designee.

2.43. "Solid and Chemical Materials" means soils, sediments, sludges, solid waste, drill cuttings, overburden, minerals, coal ash, and products and by-products of an industrial process that result in a matrix that is not otherwise defined.

2.44. "Standard Operating Procedure" means a written document that provides detailed instructions for the performance of all aspects of test analyses, operation, or action.

2.45 "Suspension of certification" means the temporary removal of approval to perform analyses under this rule until such time as the basis for suspension is rectified.

2.30. "Supervisor" means that designated person responsible for the technical adequacy and quality of data for a certification category, and who possesses the qualifications required under subsection 3.7.

§47-32-3. Certification Program; Application, Procedures, and Requirements.

3.1. Requirements of Certification.

3.1.1. With the exception of those tests not normally performed in a laboratory proper, all sample analyses required by order of the Department or performed for the purpose of determining compliance with chemical, microbiological, aquatic toxicity and radiological requirements of the State's covered statutory programs must be performed in laboratories certified for this purpose pursuant to this rule. Analyses performed in laboratories not so certified shall not be accepted by the Department as being in compliance with the requirements, rules or orders of the Department. All analyses not performed in a laboratory proper must be performed by personnel under the direction of a supervisor from a certified laboratory.

3.1.2. Laboratories doing business in other states where a state certifying agency grants reciprocal certification, approval, or other authorization to laboratories located in West Virginia, and which is certified, approved or authorized by the agency of that state having primary certification responsibility under Federal programs delegated to such other state under conditions equivalent to those required by this rule, are considered to be certified for the purpose of this rule once they have complied with the provisions of Section 3.4. Laboratories doing business in other states where certification is not required, and who are not certified in

another state, may be considered for certification by following the conditions and requirements stated in Section 3.3.

3.1.3. Only laboratories certified pursuant to this rule or maintained by the EPA may be called West Virginia Certified Environmental Laboratories and no laboratory may adopt any name or make any oral or written statement intended or likely to mislead the public with respect to its certification status.

3.2. Categories of Certification. -- A laboratory applying for certification in one or more of the following categories must demonstrate acceptable performance on proficiency test samples for all matrices, where available, and meet all other requirements of this rule. The laboratory certificate, including the list of certified parameters, will specify the categories and the parameters within each category for which the laboratory is certified and it must be displayed in a location visible to the public. Tests for all categories, except Aquatic Toxicity, must be conducted in accordance with the method and procedures specified in the Code of Federal Regulations, Chapter 40 as applicable or other methods that may be approved by EPA or the Secretary. The certification categories are:

- 3.2.1. Nonpotable Water Trace Metals;
- 3.2.2. Nonpotable Water Inorganic Nonmetals;
- 3.2.3. Nonpotable Water Volatile Organic Chemicals;
- 3.2.4. Nonpotable Water Extractable and Semi-volatile Organic Chemicals;
- 3.2.5. Nonpotable Water Dioxin and Dibenzofuran;
- 3.2.6. Nonpotable Water Microbiology, comprising tests for Coliform Bacteria, Fecal Streptococci, Pathogenic Bacteria, Plate counts, Viruses, Parasites and Parasite ova;
- 3.2.7. Whole Effluent Toxicity, testing which must be conducted in accordance with the methods and procedures specified in 40 CFR 136;
- 3.2.8. Nonpotable Water Radiochemistry;
- 3.2.9. Hazardous Waste Characteristics, including Corrosivity, Ignitability, Reactivity, Extraction Procedure Toxicity, and Toxicity Characteristic Leaching Procedure, or other tests or analyses designated by the Secretary;
- 3.2.10. Solid and Chemical Trace Metals;
- 3.2.11. Solid and Chemical Inorganic Nonmetals;
- 3.2.12. Solid and Chemical Volatile Organic Chemicals;
- 3.2.13. Solid and Chemical Extractable and Semi-volatile Chemicals;
- 3.2.14. Solid and Chemical Dioxin and Dibenzofuran;
- 3.2.15. Solid and Chemical Microbiology; and

3.2.16. Solid and Chemical Radiochemistry.

3.3. Application Procedures and Requirements for Laboratories Located in West Virginia.

3.3.1. A person operating a laboratory in West Virginia who wants to be certified in one or more of the categories and parameters thereof or, who if already certified, wants to add a category or a parameter within a category, must apply for certification to the West Virginia Department of Environmental Protection, Quality Assurance Program, refer to subsection 1.9 for the address. The applicant shall submit the appropriate fee with the application for certification.

3.3.2. An application for certification is acceptable when a complete application is submitted. This includes the appropriate fee, and the information requirements of this rule for the category, categories or parameter(s) for which certification is requested. Acceptance of a complete application does not authorize the laboratory to perform analyses regulated by this rule. The applicant will be notified if the application is not acceptable and the laboratory inspected to determine if it is in compliance with the requirements of this rule prior to the issuance of certification.

3.3.3. An application will be rejected without prejudice for not being a complete application.

3.3.4. Proficiency test samples will be an element of the laboratory evaluation. Proficiency testing will be in accordance with subsection 3.10. The laboratory must receive acceptable scores on two separate proficiency test studies prior to an on-site inspection being performed. Certified laboratories that desire to include additional parameters within previously certified categories must demonstrate satisfactory results for proficiency test samples for these additional parameters.

3.3.5. The results of the analysis of proficiency test samples shall be considered in determining whether the certification of the laboratory should be granted, renewed, denied, revoked, or suspended. Certification may be granted only for those parameters for which the laboratory performs acceptably.

3.3.6. An applicant for certification who either does not perform acceptably on the proficiency test samples or does not otherwise meet the requirements of this rule shall be notified that the requirements for certification have not been met.

3.3.6.a. Applicants receiving a notification that certification requirements have not been met may not reapply for certification until the laboratory assures the Quality Assurance Office in writing that corrective actions have been taken and documented that bring the laboratory into compliance with this rule.

3.3.6.b. Owners, principal officers, managers or supervisors of a laboratory, for which certification has been denied, may not reapply for certification of this same facility by simply changing the company or laboratory name.

3.3.6.c. Certification is transferrable. A laboratory facility must notify the Department in writing at the address listed in subsection 1.9 that the facility is being sold or has a change of principal officer(s), manager(s) or supervisor(s) within 30 days of the change or activity.

3.3.7. Certifications may contain conditions requiring correction of minor deficiencies identified by the Quality Assurance Officer by a date or dates specified therein.

3.4. Application Procedures and Requirements for Laboratories Not Located in West Virginia.

3.4.1. Owners of laboratories located in a state other than West Virginia, which have been certified, approved or otherwise authorized by that state's agency having primary certification, approval or authorization responsibility for laboratory certification programs with conditions equivalent to those required by this rule, and who have entered into a reciprocity agreement with West Virginia, and who wish to perform analyses covered by this rule for West Virginia clients shall:

3.4.1.a. Annually complete the application form provided by the Department's Quality Assurance Office;

3.4.1.b. Have the form certified by the state agency having primary certification authorization/enforcement responsibility; and

3.4.1.c. Return the form to the Quality Assurance Office of West Virginia at the address listed in subsection 1.9.

3.4.2. The application will be reviewed and if found to be complete the laboratory will be certified or recertified.

3.4.3. If the laboratory's certification, approval or authorization is revoked by the state agency having primary certification, approval or authorization responsibility, the West Virginia certification is automatically canceled for the same parameter(s) as has been revoked in the other state. The laboratory manager shall notify the West Virginia Quality Assurance Office and all clients in West Virginia of the revocation within 48 hours of receipt of notice of revocation.

3.4.4. The owner of a laboratory in a state other than West Virginia which is not certified by that state or is certified under conditions not equivalent to those required by this rule and who wishes to perform analyses for West Virginia clients may apply for certification in accordance with the procedure set forth in subsection 3.3 of this rule. In addition, prior to conducting the on-site laboratory inspection, the laboratory shall submit to the Quality Assurance Office a per diem sum the Department determines to be sufficient to cover the travel, room, and board expenses of the certification inspector(s).

3.5. Renewal of Certification. -- Applications for renewal of certification must be submitted, on forms provided therefore, no later than 180 days before the expiration date of certification, and accompanied by the appropriate fee. A laboratory submitting an application for renewal of certification may continue to operate under the previous certification until the Quality Assurance Office notifies the laboratory of the approval or denial of renewal.

3.6. Fees.

3.6.1. Owners of Laboratories applying for certification or renewal of certification, shall submit the appropriate fee obtained from the annual fee schedule specified in Table 1 for each category in which the laboratory seeks certification for one or more parameters, along with the required application materials. Fees are nonrefundable.

3.6.2. Laboratories owned or operated by the State of West Virginia or an agency of the Federal Government are exempt from the above fees, except in situations addressed in paragraph 3.6.2.a, but shall make appropriate application for certification in accordance with the other provisions of this rule.

3.6.2.a. In situations where a laboratory under this subdivision is conducting analyses for a fee,

an appropriate certification fee will be assessed.

3.6.3. All application fees collected under this rule will be paid into a special state treasury fund designated the "Environmental Laboratory Certification Fund" which will be used to defray the cost of administering this rule.

3.7. Required Laboratory Personnel Qualifications.

3.7.1. Each laboratory must have one individual designated as the person responsible or in charge and irrespective of any local title or designation, is herein referred to as the laboratory manager.

3.7.2. Current employee records must include a resume documenting each employee's training, degrees held, experience, duties, and date(s) of relevant employment. This provision is applicable only to the employee's laboratory and environmental sampling work history. Table 2 lists the minimum education and experience requirements.

3.7.3. Laboratory supervisors who are also laboratory technicians and who do not have the required laboratory experience will be considered a Supervisor-in-Training and must have their work reviewed by an individual meeting the above education and experience requirements for supervisors.

3.7.4. Technicians holding a West Virginia Environmental Training Center Wastewater Laboratory Technician certificate meet the education and experience requirements of this rule only in the conduct of analyses while employed at a Publicly Owned Treatment Works (POTW).

3.8. Duties and Responsibilities of Laboratory Personnel.

3.8.1. The laboratory manager or his designee will administer the operations of the laboratory including the approval of test and analytical results.

3.8.2. Each laboratory supervisor shall provide personal and direct supervision for personnel and for the reporting of test and analytical results.

3.9. Management of Laboratories.

3.9.1. A certified laboratory may offer as a service those laboratory tests, analyses, or procedures that are within the category or categories for which it is certified.

3.9.2. A laboratory that is certified shall only report test and analytical data for samples which are properly labeled, and for which there is reasonable assurance the samples have been collected, preserved, stored and transported in such a manner as to assure identity, stability of the sample, and proper analysis.

3.10. Proficiency Testing.

3.10.1. Except when determined by the Quality Assurance Office that an appropriate performance evaluation test is not readily available, all certified laboratories or laboratories seeking certification shall participate in a proficiency testing program covering all tests, matrices, and analyses made available within the category, categories or parameter(s) for which the laboratory is certified or seeks certification. The laboratory must participate in two studies per certification year at a frequency of one study every six (6) months.

3.10.2. Each certified laboratory or laboratory applying for certification must obtain proficiency test samples from an approved provider for each parameter and matrix for which certification is requested. The list

of approved providers is located at <http://www.a2la.org/dirsearch/nelacptproviders.cfm>.

3.10.3. Laboratories certified or those seeking certification must test or analyze the proficiency test samples and submit the results to the Quality Assurance Office or its authorized agent, as appropriate, within the time frame allowed each participant testing that set of samples for evaluation.

3.10.3.a. A laboratory may not send proficiency test samples to another laboratory for testing.

3.10.3.b. A laboratory shall not knowingly receive proficiency test samples from any laboratory seeking certification or certified by this office.

3.10.3.c. A laboratory shall not discuss proficiency test sample data with any other laboratory for any purpose.

3.10.3.d. Any laboratory found in violation of 3.10.3.a, 3.10.3.b, or 3.10.3.c will be denied certification and not allowed to reapply for certification for a period of five (5) years from the date of the denial.

3.10.4. The laboratory will have satisfied the requirements for testing for a parameter when it receives an 'Acceptable' evaluation for that parameter, in two of the last three proficiency test studies.

3.10.5. The laboratory will be informed of the results of each evaluation by the proficiency test provider. For those parameters which a laboratory has not successfully completed the proficiency test after three attempts, the laboratory will be reevaluated upon written request.

3.10.6. Acceptance limits for proficiency tests will be established according to the USEPA document "National Standards for Water Proficiency Testing, Criteria Document." For analytes and matrices not found in this document, limits will be established in accordance with the procedures set forth by the current National Environmental Laboratory Accreditation Conference (NELAC).

3.10.7. The laboratory will have three separate opportunities to acceptably analyze proficiency test samples for any parameter for which the laboratory seeks certification. The laboratory need only repeat proficiency tests for those parameters for which the laboratory has failed to perform acceptably. Parameters for Organic Samples shall mean a method, or method subdivision (i.e. Volatiles, Extractables, BTEX, etc.). Laboratories that fail to successfully analyze two of three different sets or rounds of proficiency test samples in the time period allotted will not be certified for the failed parameters until two consecutive sets or rounds have been successfully analyzed.

3.10.8. This rule incorporates by reference the 2003 National Environmental Laboratory Accreditation Conference (NELAC) Proficiency Testing standard, Chapter 2 with appendices for the purposes of Proficiency Testing Criteria for Laboratory Certification.

3.11. Laboratory Inspections.

3.11.1. As a condition of obtaining and maintaining certification, a laboratory will permit and facilitate inspections by personnel of the Department. This inspection will include the physical facilities as well as laboratory records and reports.

3.11.2. The Department will conduct at least one on-site inspection of a laboratory seeking certification to determine whether or not the laboratory meets the Quality Assurance Office standards as set forth in

this rule.

3.11.3. Regular inspections of laboratories certified in accordance with this rule will be conducted during reasonable hours. These inspections will be conducted annually or as determined by the Secretary, however, in no situation may more than two years elapse between inspections.

3.11.4. Authorized representatives of the Department may make inspections of a certified laboratory whenever the Department in its discretion considers such inspections necessary. A laboratory's refusal to allow entry to the Department's representative will be grounds for denial or revocation of certification.

3.11.5. During inspections, consideration will be given to staff competence, working conditions, tests or analytical methods used, quality control procedures, quality assurance programs, maintenance of records and compliance with the requirements of this rule.

3.11.6. The laboratory will be furnished with a copy of the inspection report which will list deficiencies found.

3.12. Cancellation, Suspension, and Revocation of Certification.

3.12.1. Any certified laboratory may cancel its certification in any category or parameter by notifying the Quality Assurance Office in writing of the laboratory's decision to cancel its certification. This cancellation notification will not entitle the laboratory to any refund of fees paid.

3.12.1.a. If the laboratory wishes to cancel the entire certification, all categories and parameters, the laboratory will enclose its Environmental Laboratory Certificate with the letter of notification.

3.12.2. A laboratory's certification may be suspended for failure to correct deficiencies within the specified timeframe.

3.12.3. A laboratory's certification may be suspended for failure to correct proficiency test sample failures.

3.12.4. A laboratory's certification may be revoked if the laboratory commits any falsification relating to certification, testing, or reporting of analytical results or for failing to comply with the provisions in 3.10.

3.13. Effect and Duration of Suspension and Revocation.

3.13.1. The results of any tests or analyses performed after the effective date of a suspension or revocation order for any category or parameter will not be accepted by the Department as compliance with the requirements of the Covered Statutory Programs as defined in subsection 2.20.

3.13.2. Suspension or revocation will not be withdrawn until the basis for the suspension or revocation has been eliminated or rectified.

3.13.3 Any laboratory having its certification suspended or revoked must notify all clients of the suspension or revocation.

3.14. Notice of Changes -- In the event there are any changes in the name, location, ownership, address, telephone number or supervisory personnel of the laboratory to which the provisions of this rule apply, then the

laboratory will immediately submit written notice thereof to the Department. For supervisory personnel this provision applies only to those whose responsibilities include analyses that must be made in compliance with this rule.

§47-32-4. Laboratory Requirements.

A certified laboratory or a laboratory seeking certification must continually meet and follow the requirements of this section.

4.1. Laboratories will have on the premises and under the control of the laboratory manager all of the equipment and instruments necessary to analyze each parameter in which the laboratory is certified, or is seeking certification. All equipment must meet the minimum standards required by the test method used.

4.2. General Requirements for All Laboratories.

4.2.1. Adequate laboratory space and facilities, to include equipment and instruments must be available to properly carry out the services performed in the laboratory.

4.2.2. Laboratory work areas will be arranged so as to minimize problems in contamination, transportation and communication.

4.2.3. Workbench space within the laboratory must be ample for the tests or analyses to be performed, have adequate lighting and be convenient to a sink, water, gas, vacuum and electrical outlets or other utilities as necessary to properly carry out the specific tests or analyses to be performed.

4.2.4. Temperature and humidity within the laboratory are to be maintained within the limits required for the proper performance of each test or analysis, the proper operation of the various instruments, and the proper storage of expendable supplies.

4.2.5. pH meters must have an accuracy of and scale graduations within 0.1 standard unit.

4.2.6. Analytical and pan balances are to be clean, not corroded, and be provided with Class-S weights or equivalent. Analytical balances will be capable of weighing to 0.1 milligram minimum. Pan balances will be capable of weighing to 100 milligrams.

4.2.6.a. An analytical balance must be mounted on a heavy, shockproof table. The balance level must be checked each use and adjusted as necessary;

4.2.6.b. An analytical balance must be located in an area that is not near laboratory traffic and is protected from drafts and humidity changes; and

4.2.6.c. Three Class-S or equivalent weights are to be available for checking the analytical balance. These weights must cover the range expected to be encountered during routine analyses.

4.2.7. All temperature measuring devices will be graduated in one degree Celsius (or 2 degrees Fahrenheit) increments and readable to 0.5 degrees Celsius (1 degree Fahrenheit) for all analyses except fecal coliform analysis; in which case glass or metal thermometers are to be readable to 0.2 degrees Celsius.

4.2.7.a. Continuous temperature recording devices will be sensitive and accurate to within 1.0 degree Celsius (2 degrees Fahrenheit).

4.2.7.b. The column of liquid in glass thermometers will have no separation.

4.2.7.c. Liquid column in glass and electronic type thermometers without a current manufacturer's certificate of accuracy must be verified as accurate annually. All other types, to include Automatic Temperature Compensation (ATC) devices, must be verified as accurate quarterly. Verification must be accomplished by comparison to a certified thermometer traceable to a National Institute for Standards Testing thermometer. See also paragraph 5.2.2.g for additional thermometer requirements.

4.2.7.d. Each temperature measuring device must be uniquely identified. The results of accuracy verifications must be documented. The corrected temperature must be recorded whenever temperatures are required to be recorded.

4.2.8. Sample storage refrigerators must maintain an internal temperature of ≤ 6 degrees Celsius.

4.2.9. Laboratory glassware, plastic ware, and metal utensils will meet the following requirements:

4.2.9.a. Glassware and metal utensils must resist corrosion, and be capable of withstanding high temperatures, and vigorous cleaning;

4.2.9.b. Flasks, beakers, dilution bottles, culture dishes, culture tubes and other glassware are to be of borosilicate glass and free of chips, cracks, and excessive etching;

4.2.9.c. Volumetric glassware must be Class A and need not be calibrated before use. Non Class A glassware must be calibrated before use; and

4.2.9.d. Metal utensils must be made of stainless steel or other inert material.

4.2.10. Pipettes must meet the following requirements:

4.2.10.a. Glass pipettes are to be made of borosilicate glass;

4.2.10.b. Plastic pipettes must be compatible with the reagents being measured, i.e. will not dissolve or show signs of etching or numbers being removed;

4.2.10.c. Plastic pipettes must be sterile or sterilizable for microbiological procedures;

4.2.10.d. Pipettes must deliver the required volume quickly and accurately within a 2.5 percent tolerance; and

4.2.10.e. Pipettes must not be excessively etched, nor the mouthpiece or delivery tips chipped, or the graduation marks illegible.

4.2.11. Magnetic stirrers must have variable speeds, and use Teflon coated stirring bars.

4.2.12. Volumetric dispensing devices including autopipetors, autotitrators and digital burets must be of sufficient sensitivity for the application. Delivery volumes of mechanical volumetric dispensing devices must be checked using the gravimetric method or using Class A volumetric glassware one every 3 months.

4.2.13. All purchased reagents and solutions must be certified as appropriate for the intended use by

the manufacturer or supplier or must be verified as appropriate by the laboratory prior to use.

4.3. Criteria and Procedures for Trace Metal Testing.

4.3.1. The Department incorporates methods approved in 40 CFR §136.3 Table IB, the current approved edition of EPA publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, and other methods as may be approved by EPA or the Secretary, including all standards, criteria, sample collection procedures, analytical procedures, methodology, quality assurance and quality control specifications for evaluation and certification purposes.

4.4. Criteria and Procedures for Inorganic Nonmetals.

4.4.1. The Department incorporates methods approved in 40 CFR §136.3 Table IB, the current approved edition of EPA publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, and other methods as may be approved by EPA or the Secretary, including all standards, criteria, sample collection procedures, analytical procedures, methodology, quality assurance and quality control specifications for evaluation and certification purposes.

4.5. Criteria and Procedures for Volatile Organic Chemicals, Extractable and Semi-volatile Chemicals and Dioxin and Dibenzofuran.

4.5.1. The Department incorporates methods approved in 40 CFR §136.3 Table IC, ID and IG, the current approved edition of EPA publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, or such other methods as may be approved by EPA or the Secretary, including all standards, criteria, sample collection procedures, analytical procedures, methodology, quality assurance and quality control specifications for evaluation and certification purposes.

4.6. Criteria and Procedures for Microbiological Testing.

4.6.1. The Department incorporates from methods approved in 40 CFR §136.3 Table IA, or other methods as may be approved by EPA or the Secretary, including all standards, criteria,⁴ sample collection procedures, analytical procedures, methodology, quality assurance and quality control specifications for evaluation and certification purposes.

4.6.2. Laboratory pure water for use in microbiological examinations will be analyzed for the parameters listed in Table 3. Should the test results for any of the substances exceed the standards set forth in the table, corrective action must be taken and the water retested.

4.6.2.a. Analysis of laboratory pure water for use in microbiological examinations must be performed by a laboratory certified under this rule. Results must be maintained and include the date, type of analysis, results and identity of the individual responsible for the results.

4.6.2.b. For purchased laboratory pure water for use in microbiological examinations, a current certificate of analysis from the producer documenting that the purity of the water is traceable. The purchased laboratory pure water must meet the requirements of Table 3.

4.7. Criteria and Procedures for Whole Effluent Toxicity Testing.

4.7.1. All work is to be performed in accordance with procedures outlined in Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, EPA/821/R-

02/012, or Short Term Methods for Estimating Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, EPA/821/R-02/013 and other methods as may be approved by EPA or the Secretary for the test to be performed.

4.8. Criteria and Procedures for Radiochemistry Testing.

4.8.1. The Department incorporates methods approved in 40 CFR §136.3 Table IE, the current approved edition of EPA publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, or other methods as may be approved by EPA or the Secretary, including all standards, criteria, sample collection procedures, analytical procedures, methodology, quality assurance and quality control specifications for evaluation and certification purposes.

4.9. Criteria and Procedures for Characteristics Testing.

4.9.1. The Department incorporates the current approved edition of EPA publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, and other methods as may be approved by EPA or the Secretary, including all standards, criteria, sample collection procedures, analytical procedures, methodology, quality assurance and quality control specifications for evaluation and certification purposes.

§47-32-5. Methodology, Quality Control and Record Keeping.

5.1. Methodology.

5.1.1. Sample collection, handling, and preservation techniques specified in 40 CFR §136.3 Table II, or other procedures approved by EPA or the Secretary are to be followed.

5.1.1.a. Samples requiring preservation will be preserved in accordance with 40 CFR §136.3 Table II for compliance with subsection 2.19.1 and the NPDES. All other samples will be preserved in accordance with applicable methods and regulations.

5.1.1.b. Sample collection, handling and preservation techniques specified by the analytical methods will be followed for the parameters analyzed by those methods in the absence of guidance under paragraph 5.1.1.a.

5.1.1.c. The chain of custody form must be completed at the time of sample collection and will state the sampling location, date and time of collection, collector's name, type(s) of preservation, number of containers per sample, type of sample (grab or composite) and any remarks.

5.1.1.d. After the sample has been collected, the appropriate information as to identity of the sample is to be written on the label. The identity of the sample must be the same on the label and the chain of custody form. The label must remain affixed to the sample container and is not to be removed until the required analyses have been completed and the surplus sample has been discarded.

5.1.1.e. The chain of custody must accompany the sample at all times. Custody of the sample must be documented on the chain of custody throughout the life of the sample (from collection to disposal of surplus sample after all required analyses have been completed). Any time the custody of the sample is transferred from one person to another, except analysts in the same laboratory, this transfer must be documented in the appropriate fields on the chain of custody form.

5.1.1.f. Immediately upon delivery of the sample to the laboratory, the individual delivering the

sample will complete the appropriate section(s) of the chain of custody form. A chain of custody form is not required where the sampler is also the analyst and in situations where the laboratory and the sample site(s) are within the property boundaries of the facility in which the laboratory is located.

5.1.1.g. Prior to accepting custody of a sample, laboratory personnel must be reasonably assured that the sample has met the chemical and temperature preservation requirements. If the sample fails to meet these requirements, the sample chain of custody form is to be marked indicating the sample was improperly preserved. Analytical data resulting from improperly preserved samples must be accompanied by a statement indicating the condition of the sample upon receipt by the laboratory. Analytical data resulting from samples improperly preserved will not be accepted as being in compliance with this rule.

5.1.1.h. When it is necessary to send samples by mail, bus, courier service, or private shipping, the chain of custody form is to be completed by the individual relinquishing custody of the sample for shipping and is to accompany the samples during shipping. Upon receipt of the samples in the laboratory, the provisions of paragraph 5.1.1.g are to be followed.

5.1.2. Test procedures identified in 40 CFR §136.3, EPA publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods or other methods approved by EPA or the Secretary will be utilized for the analysis of all samples required to be reported to the Department.

5.1.2.a. All procedures other than those set forth in subdivision 5.1.2 are considered alternate test procedures (ATP). Laboratories must make special application to the Department for the use of ATPs in accordance with 40 CFR §136.4.

5.1.2.b. All laboratories which have previously been granted approval to use an ATP by the EPA will be allowed to continue using such method after submitting written proof of the approval to the Department.

5.1.3. General Laboratory Practices.

5.1.3.a. Chemistry – Inorganic Nonmetals and Trace Metals.

5.1.3.a.A. Laboratories utilizing visual comparison devices must calibrate the standards incorporated into devices of this type at least once every four months. The laboratory will make and maintain records of the date and method of each calibration.

5.1.3.a.B. Distilled and deionized water is to have a resistivity value ≥ 0.5 megohms-cm at 25 degrees Celsius.

5.1.3.a.C. Analytical Reagent grade chemicals should be used for most analyses. Detailed information on reagent grades is set forth in the approved analytical methods and their recommendations must be followed for the reagent quality to be used for each test or analysis.

5.1.3.a.D. Where applicable, method detection limits must be determined for all categories and parameters. The method found in 40 CFR Part 136, Appendix B must be used for this calculation.

5.1.3.a.E. Field blanks must be prepared and analyzed for the test categories and parameters identified in subdivisions 3.2.1 and 3.2.2, at a minimum of two times per year, once during the cold wet season and once during the warm dry season.

5.1.3.a.F. Field duplicates must be collected and analyzed for the test categories and parameters identified in subdivisions 3.2.1, 3.2.2, 3.2.10, and 3.2.11, at a minimum of two times per year, once during the cold wet season and once during the warm dry season.

5.1.3.b. Microbiology.

5.1.3.b.A. All practices and procedures for the conduct of microbiological examinations must follow the guidance in methods approved in 40 CFR §136.3 Table IA.

5.1.3.b.B. The temperature of incubators, water baths and heat blocks must be monitored in accordance with approved methods or as specified by regulation.

5.1.3.b.B.1. Each incubator, water bath or heat block must have a thermometer placed so as to give a representative temperature measurement for the device.

5.1.3.b.B.2. Incubators, water baths and heat blocks must be clean and properly maintained in accordance with the manufacturer's instructions.

5.1.3.b.C. Autoclaves must meet the specified temperature tolerances in the approved method. The use of a pressure cooker is not recommended.

5.1.3.b.C.1. A continuous temperature recording device or a maximum temperature registering thermometer must be used to measure the temperature during each autoclave cycle.

5.1.3.b.C.2. The laboratory must use a sterilization verification technique such as autoclave tape to indicate proper sterilization of equipment and contaminated materials.

5.1.3.b.C.3. Autoclaves must be clean and properly maintained in accordance with the manufacturer's instructions.

5.1.3.b.C.4. Autoclaves must be serviced annually by a qualified person. Servicing must include a pressure check and calibration of temperature devices.

5.1.3.b.D. Hot air sterilization ovens must be of sufficient size to prevent crowding and constructed to give uniform sterilization.

5.1.3.b.D.1. Hot air sterilization ovens must be clean and properly maintained according to the manufacturer's instructions.

5.1.3.b.E. Appropriate optical counting equipment must be used in accordance with approved methods.

5.1.3.b.F. Appropriate inoculating equipment must be used in accordance with approved methods.

5.1.3.b.G. Appropriate membrane filters, pads and dishes must be used in accordance with approved methods.

5.1.3.b.H. A sterility blank must be analyzed:

5.1.3.b.H.1. with each lot or batch of media, either purchased or prepared in the laboratory;

5.1.3.b.H.2. with each lot of membrane filters, pads and dishes;

5.1.3.b.H.3. with each lot or batch of sample containers, either purchased or prepared in the laboratory;

5.1.3.b.H.4. with each lot or batch of rinse/dilution water, either purchased or prepared in the laboratory; and

5.1.3.b.H.5. with each lot or batch of culture tubes, either purchased or prepared in the laboratory.

5.1.3.b.I. Field blanks must be prepared and analyzed for the test categories and parameters identified in subdivision 3.2.6, at a minimum of two times per year, once during the cold wet season and once during the warm dry season.

5.1.3.b.J. Field duplicates must be collected and analyzed for the test categories and parameters identified in subdivision 3.2.6, at a minimum of two times per year, once during the cold wet season and once during the warm dry season.

5.1.3.b.K. All equipment and reagents must be sterilized prior to use. All contaminated equipment must be sterilized prior to reuse. All contaminated material must be rendered innocuous prior to disposal.

5.1.3.c. Whole Effluent Toxicity Testing.

5.1.3.c.A. Natural or artificial sources of water may be used, but natural sources are preferred.

5.1.3.c.B. Natural sources are to be free of pollution, low in turbidity, high in dissolved oxygen, low in B.O.D., and the pH must be favorable to the maintenance of the organisms.

5.1.3.c.C. Municipal water supplies are acceptable. Water from a municipal source must be passed through a filter to remove organic chemicals and chlorine before use, and be conditioned for the species under test.

5.1.3.c.D. Test organisms are to be fed as outlined in the approved methods, subdivision 3.2.7.

5.1.3.c.E. Treatment of diseased or parasitized organisms is to be in accordance with the procedures given in the approved methods, subdivision 3.2.7.

5.1.3.c.F. Organisms treated for disease or parasites are not to be used in whole effluent toxicity tests for at least 10 days after treatment.

5.1.3.d. Radiochemistry.

5.1.3.d.A. Analytical reagent grade (AR) chemicals will be used for all analyses, unless otherwise required for an individual analytical procedure.

5.1.3.d.B. Radioactive standards and radioactive wastes are to be stored in an enclosed and properly labeled area, either within the laboratory or in a separate room or facility. All radioactive materials must be safely stored in suitable containers.

5.1.3.d.C. Standards and samples are to be prepared in an area of the laboratory specifically designated for and exclusively used for the preparation of radioactive standards and samples. Adequate precautions must be taken in this area to ensure against radioactive contamination.

5.1.3.e Volatile Organic, Extractable, and Semi-volatile Organic Testing. Equipment must be capable of meeting the quality control requirements specified in subdivision 5.2.6.

5.1.3.e.A. Trip blanks must be prepared, transported and analyzed for each batch of samples for analysis for Nonpotable Volatile Organic Chemicals, subdivision 3.2.3.

5.1.3.e.B. A method blank must be analyzed with each batch of samples.

5.1.3.e.C. A laboratory control sample must be analyzed with each batch of samples.

5.1.3.e.D. A matrix spike and a matrix spike duplicate must be analyzed with each batch of samples. In situations where the laboratory does not receive sufficient sample volume or quantity to perform a matrix spike and a matrix spike duplicate, a laboratory control sample and a laboratory control sample duplicate must be analyzed.

5.1.3.e.E. Surrogate spike compounds must be added to all samples and quality control standards prior to preparation/extraction and analysis where applicable. The recovery of surrogate compounds must be compared to acceptance limits established in the appropriate method. If acceptance limits are not provided in the method, the laboratory must use appropriate procedures to establish in-house acceptance limits.

5.1.3.e.F. Any time criteria are not met with respect to blanks, laboratory control samples, matrix spikes, matrix spike duplicates, or surrogates, data must be reported with appropriate qualifiers describing the situation and explaining the effect on the results.

5.2. Quality Control Programs -- Each laboratory will develop, and have on file available for inspection a written description of the current laboratory Quality Assurance Program Plan. This written description will outline the procedures the laboratory uses in meeting the quality control requirements set forth in this subsection. Managers, supervisors, and analysts should participate in developing the Quality Assurance Program Plan. Each participant within the laboratory is to have access to a copy of the quality control program Quality Assurance Program Plan and the detailed guidelines for implementation of the participant's responsibility. A record of analytical control tests and quality control checks on media, materials, and equipment will be prepared by the laboratory and retained for at least three years.

5.2.1. A written description includes, but need not be limited to, the following for each category:

5.2.1.a. Procedures which the laboratory will use in meeting the quality control requirements of this rule pertaining to laboratory equipment and instrumentation, and the frequency with which these procedures will be performed.

5.2.1.b. Each laboratory will develop and maintain a written standard operating procedure (SOP) manual, which sets forth, in detail, the methods the laboratory will use in chemical analyses or tests for all parameters for which the laboratory is seeking certification.

5.2.1.c. Each laboratory must record and retain all raw data and calculations derived from analyses and quality control procedures in a manner that will provide easy verification of the data and calculations during on-site inspections.

5.2.2. Laboratories conducting analyses for Inorganic Nonmetals and Trace Metals must perform the following internal quality control checks:

5.2.2.a. Each analytical balance, with the exception of electronic balances without internal calibration controls, is to be checked and adjusted annually by a balance service technician. The accuracy of each analytical balance must be checked on each day of use using at least three Class-S weights covering the range expected to be encountered during routine analysis. The weights used, weight detected, dates on which checks were performed, analyst, record of balance level check and other pertinent information is to be recorded in a log book. The daily weighing check will be used as an indication of proper operation of electronic balances.

5.2.2.b. The accuracy of the wavelength setting of spectrophotometers without built-in automatic system diagnostics is to be checked yearly by comparing the wavelength setting to the absorption maxima appropriate standards. Any observed variation of the wavelength setting from the expected value must be within the manufacturer's stated tolerance for the instrument. The check data must be recorded in a logbook.

5.2.2.c. pH meters are to be calibrated prior to use with two pH buffer standards bracketing the value to be measured and the calibration recorded. Records of pH meter standardization must be maintained in a laboratory notebook that documents the date of standardization, calibration buffers used and the initials of the individual conducting the standardization. If the meter displays a slope or other indicator of performance, this information must also be recorded.

5.2.2.c.A. Aliquots of standard buffers may not be used for longer than one day.

5.2.2.d. The linearity of conductivity meters must be checked over the range of the instrument using at least five concentrations of standard solutions yearly. The cell constant, k , is to be determined from this data. The meter must be calibrated using at least one standard with each use. The results of these calibrations must be recorded in a log book.

5.2.2.e. A daily record of the drying oven temperature must be maintained for each day on which the drying oven is in use. The oven thermometer must be kept in a sand bed or other inert material.

5.2.2.e.A. The oven temperature must be recorded immediately prior to placing samples in the oven and then again immediately prior to removing samples at the end of the drying cycle.

5.2.2.f. The temperature of each refrigerator and each incubator is to be either recorded continuously or recorded daily from in-place thermometers immersed in liquid and placed on one of the shelves being used. The refrigerator thermometer must be kept in a low vapor pressure liquid such as 50/50 water/Ethylene Glycol.

5.2.2.g. The accuracy of all thermometers used to monitor temperatures will be verified by

comparing the readings of such thermometers with the readings of a certified thermometer. Refer to paragraphs 4.2.7.c and 4.2.7.d.

5.2.2.h. A calibration curve must consist of one calibration blank and 4 at least four standards to be prepared for each analysis requiring a calibration curve. This curve will be verified prior to each subsequent analysis by analyzing at least one calibration blank and one standard at or near the midpoint of the curve. These verifications are considered satisfactory if the result for the calibration blank is less than the method detection limit and the result for the midpoint standard is within 10 per cent of the expected value following vendor approved procedures for instrument calibration.

5.2.2.i. Standard curves used in the analysis of parameters in the Trace Metals category will be prepared in accordance with approved methods.

5.2.2.j. Where practicable, duplicate sample analyses are to be conducted for parameters in the Inorganic Nonmetals and Trace Metals categories to verify the precision of the method. Duplicate analyses will be performed at a frequency of 5 percent. Where less than 20 samples are analyzed at one time the analyst is to verify the precision once per analysis batch. Documentation will be made, in tabular form and on control charts, of precision testing.

5.2.2.j.A. In cases where sample results are normally below the method detection limit, precision must be determined by analysis of matrix spikes and matrix spike duplicates.

5.2.2.k. Where practicable, spiked sample analyses will be conducted to verify the accuracy of the method at the same frequency as set forth in paragraph 5.2.2.j of this rule. Documentation will be made, in tabular form and on control charts, of accuracy testing.

5.2.2.l. Where practicable, standard deviations are to be calculated and documented for all applicable measurements being conducted in the Inorganic Nonmetals and Trace Metals categories (spiked sample recoveries). Standard deviations must be documented in tabular form and on control charts.

5.2.3. Microbiology.

5.2.3.a. A start and finish membrane filter (MF) sterile control test of rinse water, media and supplies will be conducted for each sample filtration series. If the control tests indicate contamination, then all data which has been generated through tests involving the use of the contaminated materials will be rejected and the laboratory must request immediate resampling of those samples associated with the observed contamination.

5.2.3.b. When analyzing duplicate aliquots to assess precision, the same series of volumes/dilutions must be utilized for the sample and the duplicate.

5.2.3.c. The method detection limit for bacteria by the membrane filter method is defined as 1 colony /100 ml, adjusted as necessary for filtered volumes other than 100 ml.

5.2.3.d. The most probable number (MPN) test for bacteria must be carried through the "confirmed" stage for Fecal Coliform.

5.2.4. Whole Effluent Toxicity Testing -- An acceptable degree of precision for definitive toxicity tests is the 95 percent confidence level or fiducial intervals within less than ± 30 percent of the 48 hour or incipient LC50 value.

5.2.4.a. Five reference toxicant tests on each reference toxicant and species combination evaluated by the laboratory are to be performed to establish the validity of effluent toxicity data generated by bioassay laboratories.

5.2.4.a.A. After completion of the requirements in paragraph 5.2.4.a, a reference toxicant test must be performed each month in which whole effluent toxicity testing is conducted using the same method and species as used for the whole effluent toxicity testing.

5.2.4.b. Quality control and proficiency test samples are available from commercial sources.

5.2.4.c. The reference toxicant test must be conducted within 7 days immediately preceding a whole effluent toxicity test or concurrently with the whole effluent toxicity test.

5.2.4.d. A control chart, as described in approved methods, should be prepared for each reference toxicant/species combination, and successive LC-50's plotted and examined to determine if the results are within prescribed limits.

5.2.4.e. If the LC-50 of a reference toxicant does not fall in the expected range for the test organisms, the sensitivity of the test system is suspect. In this case, the test procedure should be examined for defects, and a different batch of test organisms should be employed in repeating the reference toxicant and effluent toxicity test.

5.2.5. Radiochemistry -- Permanent records must be maintained of preventive maintenance, periodic inspections, testing, and calibration for the proper operation of radiation instruments and analytical balances; validation of methods; evaluation of reagents and volumetric equipment; surveillance of results; and remedial actions taken in response to detected defects. Such records must be kept on file by the laboratory for a period of at least five years.

5.2.5.a. To verify internal laboratory precision, duplicate analyses equal to ten percent of sample analyses shall be performed. The differences between duplicate measurements shall be less than twice the standard deviation of the specific analysis as described in Environmental Radioactivity Laboratory Intercomparison Studies Program, EPA 600/4-77-001 and other guidance from EPA or the Secretary.

5.2.5.b. One background and one calibration standard must be tested each day at a 5 percent level or fraction thereof.

5.2.5.c. Work records of quantitative tests are to indicate final results together with all corresponding instrument readings and calculations. Where instrumentation produces tracings or printouts, such tracings or printouts may serve as the work record.

5.2.6. Volatile Organic, Extractable and Semi-volatile Organic Testing.

5.2.6.a. The frequency and procedures for satisfying each of the requirements listed in paragraphs 5.2.6.b and 5.2.6.c are described in detail in EPA publication SW-846, 40 CFR Part 136, and/or in the US EPA Contract Laboratory Program Statement of Work for Organics Analysis.

5.2.6.b. Minimum quality control operations necessary to satisfy the analytical requirements associated with the determination of semi-volatile and volatile organic compounds by gas chromatographic methods will include the following:

5.2.6.b.A. Evaluation of Appropriate Blank Materials.

5.2.6.b.B. Surrogate Spike Response Monitoring.

5.2.6.b.C. Matrix Spike and Duplicate Analyses or Matrix Spike Duplicate.

5.2.6.b.D. Verification of Response and Calibration.

5.2.6.b.E. Conformational Analysis.

5.2.6.c. Minimum quality control operations to satisfy the analytical requirements associated with gas chromatographic/mass spectrometry determinations of semi-volatile and volatile compounds will be as follows:

5.2.6.c.A. Documentation of GC/MS Mass Calibration and Tune Abundance Patterns.

5.2.6.c.B. Documentation of GC/MS Response Factor Stability.

5.2.6.c.C. Internal Standard Response and Retention Time Documentation.

5.2.6.c.D. Surrogate Spike Recovery Monitoring

5.2.6.c.E. Matrix Spike and Duplicate Analyses or Matrix Spike Duplicate.

5.3. Records and Data Reporting.

5.3.1. Records of analyses, including but not limited to all raw data, calculations, quality control data, and laboratory reports, are to be kept by the laboratory for at least five years unless otherwise specified.

5.3.2. The following information is to be retained by the laboratory as part of the records of analysis and the records of custody:

5.3.2.a. The laboratory number or other form of identification of the sample;

5.3.2.b. The chain of custody form as required under paragraph 5.1.1.c;

5.3.2.c. The date and time when the laboratory received the sample, whether the sample was received preserved or unpreserved;

5.3.2.d. The date and time of analysis of the sample;

5.3.2.e. The person or persons who performed the analysis;

5.3.2.f. The type of analysis performed and the analytical method or methods employed;

5.3.2.g. The raw data generated by the analysis and results of the analysis; and

5.3.2.h. The name and address of the laboratory to which the sample was forwarded, if the analysis was not performed at the laboratory which first received the sample.

5.3.3. If the chain of custody information is reported on a chain of custody form, a copy of the form must be attached to the sample report form.

5.3.4. The results of each analysis are to be calculated and entered on the sample report form which is to be forwarded to the person requesting the analysis of the sample. A careful check is to be made to assure that each result entered on the sample report form is the same as the result generated by the analysis and entered on the bench sheet or other raw data document.

5.3.5. The original or true duplicate of the results of the test or analysis is to be sent promptly to the person who requested such tests or analysis, and must be signed by the laboratory manager or a designee whose designation has been documented in the laboratory Quality Assurance Manual or other instrument describing pertains within the laboratory.

5.3.6. Whenever a laboratory subcontracts samples to another laboratory, the person ordering the examination is to receive the original laboratory report or a true duplicate of that report on the form generated by the subcontract laboratory that actually performed the test or analysis.

5.3.7. If results are entered into a computer storage system, a printout of the data must be verified with the raw data.

5.3.8. The final data report must contain the following:

- 5.3.8.a. The name, address, and contact information of the laboratory performing the analyses;
- 5.3.8.b. Sample identification number (unique identifier assigned by the laboratory);
- 5.3.8.c. Sample description;
- 5.3.8.d. Date sample was collected;
- 5.3.8.e. Date sample was received at the laboratory;
- 5.3.8.f. Date of each individual analysis;
- 5.3.8.g. Method detection limit for each parameter;
- 5.3.8.h. Identity of the test method(s);
- 5.3.8.i. Deviations from the test method, if applicable;
- 5.3.8.j. Disclosure of contract laboratory and original or true copy of the results from the contract laboratory; and
- 5.3.8.k. Identity of the responsible agent.

§47-32-6. Appeals.

Appeal to Environmental Quality Board -- Any person aggrieved or adversely affected by an order or action of the Secretary made and entered in accordance with the provisions of this rule or by issuance or denial of certification under the provisions of this rule, may appeal to the Environmental Quality Board in the same manner as appeals are taken under W. Va. Code §22B-1-7 to have the order vacated or modified. The filing of a notice of appeal will not automatically stay an order or action of the Secretary. The Environmental Quality Board will be reimbursed from the Environmental Laboratory Certification Fund for expenses incurred for appeal hearings filed with the Board relative to the provisions of this rule.

TABLE 1:

ENVIRONMENTAL LABORATORY CERTIFICATION
ANNUAL FEE SCHEDULE

Application fee – initial application.....	\$100.00
Application fee – renewal application.....	\$80.00
Application fee – additional parameters/methods	
When added other than at renewal.....	\$50.00
Nonpotable water Trace Metals – per metal – one method.....	\$20.00
Each additional method for the same metal.....	\$10.00
Nonpotable water Inorganic Nonmetals – per analyte or parameter – one method.....	\$50.00
Each additional method for the same analyte or parameter.....	\$25.00
Nonpotable water Volatile Organic Chemicals – per method.....	\$250.00
Per category maximum.....	\$750.00
Nonpotable water Extractable and Semi—volatile Organic Chemicals – per method.....	\$250.00
Per category maximum.....	\$750.00
Nonpotable water Dioxin and Dibenzofuran.....	\$1000.00
Nonpotable water Microbiology – per parameter per method.....	\$75.00
Whole Effluent Toxicity – acute.....	\$750.00
Whole Effluent Toxicity – chronic.....	\$750.00
Nonpotable water Radiochemistry.....	\$600.00
Solid and Chemical Trace Metals – per metal – one method.....	\$20.00
Each additional method for the same metal.....	\$10.00
Solid and Chemical Inorganic Nonmetals – per analyte or parameter – one method.....	\$50.00
Each additional method for the same analyte or parameter.....	\$25.00
Solid and Chemical Volatile Organic Chemicals – per method.....	\$250.00
Per category maximum.....	\$750.00
Solid and Chemical Extractable and Semi-volatile Organic Chemicals – per method.....	\$250.00
Per category maximum.....	\$750.00
Solid and Chemical Dioxin and Dibenzofuran.....	\$1000.00
Solid and Chemical Microbiology – per parameter per method.....	\$75.00
Solid and Chemical Radiochemistry.....	\$600.00
Hazardous Waste Characteristics – per procedure.....	\$150.00

TABLE 2:

EDUCATION & EXPERIENCE REQUIREMENTS
FOR SUPERVISORS

CERTIFICATION CATEGORY	EDUCATION + (Years)(1)	+	EXPERIENCE (Years)(2)	SPECIAL REQUIREMENTS
Limited Chemistry & Microbiology	12 14 16	+ + +	2 or 1 or 1	ETC Certificate(3)
Atomic Absorption	16	+	2(4)	2 years of experience must be in atomic absorption
Gas Chromatography	16	+	2(4)	2 years of experience must be in gas chromatography
Mass Spectrometry	16	+	2(4)	2 years of experience must be in mass spectrometry
Whole Effluent Toxicity	16	+	2(4)	2 years of experience must be in whole effluent toxicity testing
Radiochemistry	16	+	2(4)	2 years of experience must be in radiochemistry

Notes:

(1) 12 years = High School diploma or GED.

14 years = 2 years of college with emphasis in laboratory technology or a natural science.

16 years = Bachelors degree in Chemistry, Biology, Environmental Science, or other natural science.

(2) Substitution -- 1 year of laboratory experience within the specific certification category may be used for each year of education beyond 12 years.

(3) ETC Certificate = Environmental Training Center Laboratory Technician Certificate required of all POTW laboratory supervisors.

(4) No substitution is allowed for the 2 years of minimum experience required.

TABLE 3:

QUALITY OF PURIFIED WATER USED IN MICROBIOLOGY TESTS

Test	Monitoring Frequency	Limit
Chemical Tests:		
Conductivity	With each use	>0.5 megohms resistance or <2 umhos/cm at 25 degrees Celsius
pH	With each use	5.5 - 7.5
Heavy Metals (Cd, Cr, Cu, Ni, Pb, Zn)		
(single)	Annually	<0.05 mg/L
(total)	Annually	<0.10 mg/L
Ammonia/Organic N	Monthly	<0.10 mg/L
Total Chlorine Residual	with each use	< detection limit (0.01 mg/L maximum which ever is lower)
Total Organic Carbon	Monthly	<1.0 g/L
Bacteriological Tests:		
Heterotrophic Plate Count	Annually	<1000 colonies/mL